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Workshop on Reliability Issues in Nanomaterials

17-19 August 2004

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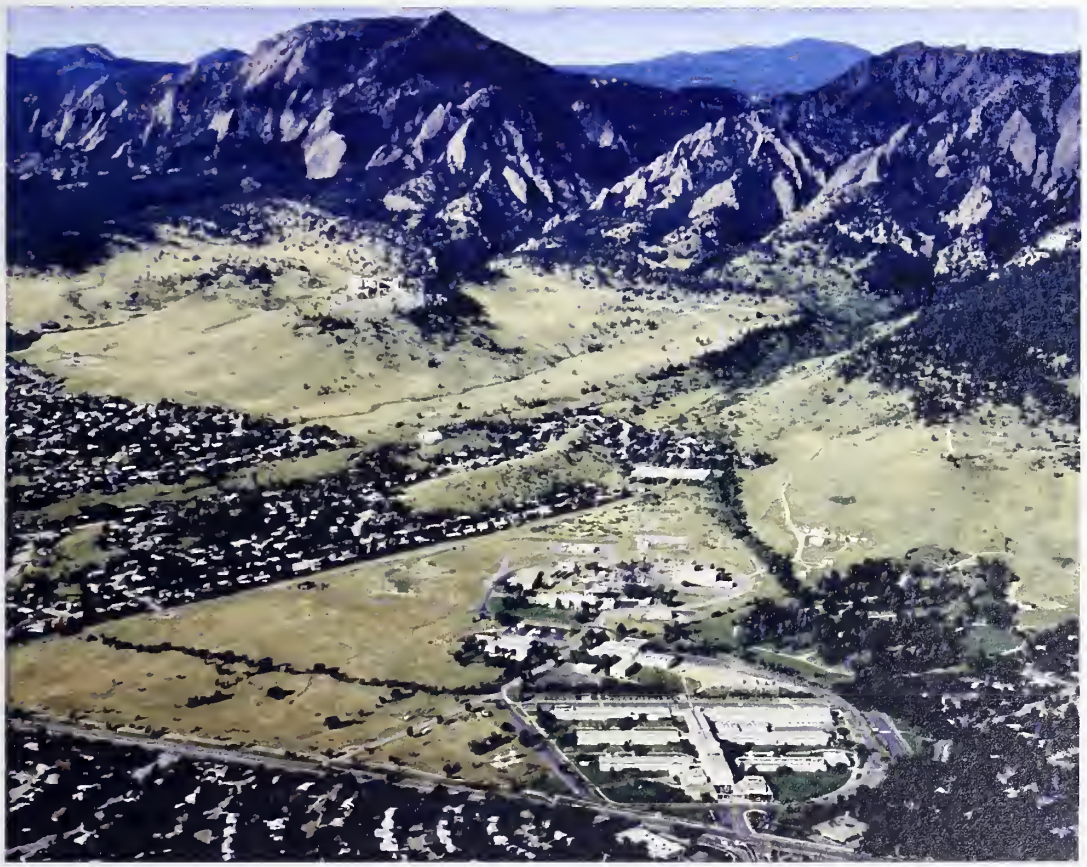

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EXECUTIVE SUMMARY

The Workshop on Reliability Issues in Nanomaterials was held at the Boulder Laboratories of the U. S. Department of Commerce on August 17-19, 2004. It was organized by the National Institute of Standards and Technology (NIST) and was designed to promote a particular subset of NIST's responsibilities under the National Nanotechnology Initiative (NNI). Larger issues, such as the definition, prospective uses, and impact of nanomaterials have been addressed elsewhere and were not considered in this workshop. The goal was to achieve consensus on two related topics: the identification of specific measurement-related barriers to successful incorporation of reliable nanomaterials into widespread engineering practice in the next 5 to 10 years; and the identification of measurement methodologies, standards, data, and models that might be appropriate for overcoming these barriers. 34 participants, representing cutting-edge nanomechanics-related research and development in industry, academia, and national laboratories, attended and contributed. Adding NIST-Boulder staff who attended brings the total to about 40. The workshop format promoted discussion on the intended topics, and included

1. a pre-workshop questionnaire to be addressed by plenary and breakout speakers;
2. invitation of 10 plenary speakers who gave hour-long presentations and 23 breakout speakers, who gave brief presentations addressing the points raised in the questionnaire.

Key Conclusions: Industrial, academic, and national laboratory consensus indicated that there is always a need to understand fundamental causes of failure. Such understanding should then lead to redesign that is more reliable and to improved manufacturing. The goal of accurate performance and lifetime prediction for nanomaterials depends on the interplay between accurate materials testing and characterization, and reliability models incorporating valid measured data. Attendees agreed that while the synergy among industry, academia, and national laboratories was effective, more fundamental materials research is needed, where the actual division of labor would be determined by market forces and policy.

It was suggested that NIST could serve the unique role of developing metrology, standards, and materials characterization methods for improving reliability of nanomaterials. The most challenging and general metrology recommendation was the development of an "atom imager," a hypothetical instrument capable of *nondestructively* measuring the chemical identity and precise three-dimensional position of every atom within a nanomaterial. Such an instrument was postulated to be the key tool for optimizing fabrication/manufacturing and controlling reliability of nanomaterials. Nearer-term recommendations centered on improving the metrological performance of scanned probe microscopy (SPM) and nanoindentation. A secondary theme in many presentations was the need for modeling to be formally coupled with physical measurement in any study of nanomaterials; however, no novel computational tool or dataset was identified as a need of high priority.

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February 2005

Workshop on Reliability Issues in Nanomaterials

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Keywords: design; experiment; fabrication; failure; lifetime prediction; material characterization; metrology; modeling; processing

1. INTRODUCTION

Nanomaterials lie at the heart of the field of nanotechnology. The NNI projects this field to have tremendous global impact, estimated to become a $1 trillion industry over the next 15 to 20 years, with one third of that focused on materials and materials processing. At present, with an estimated $1 billion annual investment, U.S. government spending has doubled in the last 4

years and constitutes nearly one third of the world governments' annual investment of over $3 billion. Through the NNI, approximately 22 % of that U.S. investment addresses nanostructured materials by design, and another 39 % addresses nano-electronics, -photonics, and -magnetics; the rest cover other NNI grand challenges such as health care, environment, energy, instrumentation and metrology, and manufacturing.

The introduction of nanomaterials into current and future technologies opens up an entirely new suite of both materials science and measurement science challenges. Effects of dimensional scaling play a stronger role in the reliability of nanomaterials than in any other materials known to date. Surfaces and interfaces can easily dominate and change behaviors and properties known to develop in bulk materials of the same chemical composition. As a result, one cannot simply extrapolate what is known about bulk material behavior to the nanoscale and expect to predict structure or properties accurately.

The full realization of reliable devices at this scale is limited by a host of materials science and engineering barriers centered on nanomechanics. For example, the development of accurate and repeatable metrologies for determining mechanical properties of materials is key to understanding how issues such as plasticity, fracture, adhesion, friction, stress/strain response, micro- and nanostructure, and chemistry determine the mechanical response of materials at the nanoscale. Further, nanomechanical response plays an important, yet sometimes indirect, role in other types of reliability, such as thermal or electrical reliability. ***Valid measurements can provide a foundation for building predictive models of nanostructure and behavior that will be based on materials science, as opposed to being purely empirical in nature.***

It was the intent of this workshop to identify and discuss specific measurement-related barriers to successful incorporation of reliable nanomaterials into widespread engineering practice in the next 5 to 10 years, as determined by leading researchers in nanomechanics and reliability. The workshop also intended to identify measurement methodologies, standards, data, and models that might be appropriate for overcoming these barriers.

This report is also available online at http://www.boulder.nist.gov/div853/RIN .

2. *SCOPE*

These topics of discussion described the workshop for invitees considering attendance:

- Nanomaterials processing and fabrication and their effects on mechanical reliability
- Methods and techniques for characterizing structure of nanomaterials
- Methodologies for measuring mechanical properties in nanomaterials
- Experimental and modeling approaches to understanding effects of structure and dimensional scaling on mechanical behavior of nanomaterials, with emphasis on surface and interface issues
- Approaches to understanding what roles nanomechanics plays in other types of reliability
- Structure, property, and lifetime prediction

Invitations to potential attendees informed them that we were seeking not only a description of the present state of the art in nanomechanical reliability, but also a glimpse into the future of the possible paths into which nanotechnology-based research could lead us in materials science and engineering, and vice versa.

This workshop addressed some of the Grand Challenges identified during the Nanomechanics Breakout Track of the NNI Interagency Workshop on Instrumentation and Metrology, held January 2004 at NIST-Gaithersburg. Namely, we addressed many of the following issues:

- Modeling of nanomechanical experiments;
- Integration of multiple techniques for measurement and characterization
- Experimentation/testing under real application conditions
- Standardization and calibration
- Instrument development for nanomechanics
- High-throughput, automated nanomechanics measurements

3. DEVELOPMENT OF NEEDS STATEMENTS

The last two hours of the workshop were used to review a two-part summary, written by the NIST organizers, of the measurement needs expressed by the attendees. One part was intended to be a complete list of specific needs expressed by all speakers, grouped according to possible metrological solutions. The other part was a shorter list of the consensus summary of the needs identified at the workshop. It became clear during the course of the workshop that materials researchers who are concerned with understanding and improving nanomechanical reliability commonly use several main tools:

- Nanoindentation
- Micro- and nanotensile and compression testing
- Scanned probe microscopy
- Modeling

A number of other important tools were discussed, for example, reflectivity measurement, for reflective coatings on mirrors intended for switching light signals to and from optical fibers, and scanned x-ray diffraction, for analysis of grain structure in copper on large silicon wafers; but the degree of convergence on nanoindentation, scanned probe microscopy, and modeling was neither intended nor anticipated in the planning of the workshop. As a result, most of the detailed and consensus need statements center around these measurement instruments.

The idea of the measurement issues important to researchers from different industries being addressed by a smaller suite of measurement tools was crystallized by one of the attendees in a breakout presentation. She said: "We care about different technologies and reliability concerns, but are addressing the same problems."

4. OUTLINE OF WORKSHOP REPORT

We present an assessment of current nanomaterials fabrication and characterization knowledge in the context of reliability, based on questionnaires and discussion during the plenary sessions, and follow with a detailed summary of the issues of concern for making reliable nanomaterials, as articulated by all workshop attendees. Plenary speakers reviewed this report prior to its distribution.

Two alternative definitions of the reliability of nanomaterials are considered, as used in the context of this workshop:

1. the extent to which nanomaterials exhibit consistent mechanical behavior and associated properties over a long period, such as during a device lifetime;
2. the extent to which nanomaterial behavior and properties can be predicted over a given period.

The first definition is similar to the conventional use of the term "reliability." However, the second definition must sometimes be used in the design of devices and products, in the event that a material changes or degrades with increasing exposure to design stresses and environments. If the material degrades over time, but the degradation can be well predicted, the material may still be useful. In general, a "reliable" nanomaterial is one that can serve its purpose over the course of an intended device or designed product lifetime.

5. ASSESSMENT OF CURRENT KNOWLEDGE IN NANOMATERIALS FABRICATION AND CHARACTERIZATION

5.1 Reliability Issues Pertinent to Nanomaterials

Successful incorporation of nanomaterials into widespread engineering use requires us to understand why, how, and when failure will occur. Basic research addressing the combination of identification and measurement of failure mechanisms in these novel materials will provide the framework necessary to develop reliability models that are able to accurately predict changes in behavior and properties, and therefore lifetime, a key factor in successful manufacturing.

A number of factors that compromise reliability were identified during the workshop. The detailed lists as articulated by attendees follow in the next section, and are summarized here. We have sorted the reliability problems into three categories:

Structures, behaviors, and resulting properties

- Nanomaterials have much larger surface-to-volume ratios than bulk materials. This affects friction and wear properties as well as stiction in nanomaterials.
- Polycrystalline nanomaterials contain a higher fraction of grain boundary volume, and may be more susceptible to failure mechanisms involving such boundaries, including

high-temperature deformation modes becoming operative at lower temperatures or failure mechanisms involving diffusion.

- Nanomaterials are almost always used as part of a materials *system*, and invariably come into contact with other materials, creating interfaces. Failure mechanisms involving delamination or chemical interdiffusion across an interface can be exacerbated. Diffusion through interfaces can also contribute to failures.
- Defect stability differs from that observed in bulk materials due to stronger influences of surfaces. Strain hardening may not follow the stages seen in bulk materials since dislocations can easily escape. Internal stresses associated with defects can build up, occupying the entire volume of a nanomaterial faster than might be expected from observations of bulk materials. Dislocations may not be energetically favored to exist in extremely small structures, forcing other mechanisms of plasticity or fracture to operate.
- Grain size is usually smaller in polycrystalline nanomaterials than in bulk materials, causing defect behavior and properties such as strength to differ from those seen in bulk materials.
- Localized behavior is much more important in nanomaterials. For example, an unusual orientation of a single grain can compromise the reliability of an electronic interconnect.

Operation under extreme conditions

- Accelerated tests must be developed in manners that suitably reproduce expected operating conditions, but sometimes the expected conditions can lead to behaviors not yet well understood in nanomaterials.
- *Area- or volume-normalized* external loads can be much more intense than in the case of bulk materials. Nanomaterials are often expected to withstand current densities, electric fields, pressures, stresses, or optical power densities that far exceed the values typically applied to bulk materials. Behaviors under such conditions are largely unknown.
- Time dependence of failure is very different from that observed in bulk materials, due to vastly different boundary conditions and length scales.
- Thermal management in nanomaterials is difficult, due to the high density of interfaces in multi-material systems. This is exacerbated by very high temperatures often undergone during thin- film or high-pressure processing.

Size, shape, and distribution control

- Ordered arrays or patterns of some nanostructures cannot presently be sufficiently fabricated. Quantum dots or carbon nanotubes must be made uniformly over large areas in order for many potential devices to be realized. Arrays and patterns must contain well controlled size and structure distributions. Lithographic approaches may not be

feasible beyond certain dimensional limits, suggesting the need for self-assembly or other patterning processes.

- Stresses and strains can become very large in nanomaterials, and their management becomes extremely difficult. Although lattice-mismatched quantum dots (QDs) require high elastic strains (~7 % for InAs/GaAs) in order to exhibit their unique electronic behaviors, plastic relaxation must be avoided.

5.2 State of the Art in Measurement Technology for Reliability Studies of Nanomaterials

Reliability studies of nanomaterials depend on valid measurement of the properties and behaviors of interest. Thorough understanding of mechanical properties and behavior of nanomaterials requires accurate knowledge of the external forces and displacements applied to such materials, as well as the corresponding response of those materials.

Many test and characterization (i.e., measurement) methods were discussed during the workshop. Some are now in use, while others are still under development. In general, high performance tools for measuring bulk materials are commonly used for nanomaterials characterization, with appropriate modifications. For example, high resolution imaging methods such as atomic force microscopy (AFM), scanning tunneling microscopy (STM), transmission electron microscopy (TEM), and scanning electron microscopy (SEM) are routinely pushed to their limits. X-ray diffraction (XRD) is also used extensively for structural characterization of both aggregates and individual nanostructures. Focused ion beam (FIB) microscopy and manipulation has become a necessity for direct imaging, preparing specimens for other types of imaging, and patterning. Advances in mechanical testing and characterization have led to unprecedented resolutions in measurement and control of force and displacement. We summarize these state of the art methods here, categorized by application. A detailed listing of testing and characterization needs exceeding the present state of the art, as articulated by attendees, follows in the next section.

MEMS/NEMS

- Nondestructive and/or noninvasive measures for mechanical displacement, both static and dynamic, using electrical or optical methods
- Automated systems for testing at high throughput
- Surface analysis and analytical tools for identifying impurities or contaminants, e.g., Auger electron spectroscopy, X-ray photoelectron spectroscopy, secondary ion mass spectroscopy (SIMS)
- Environmental chambers for accelerated testing
- Shock and vibration testing at the product level
- Thermal shock and cycling tests for delamination and crack growth

Nanoscale Manufacturing and Assembly

- STM methods for moving and removing atoms, to enable bottom-up fabrication
- Patterned atomic layer deposition (ALD) using STM for patterning (idea near realization)
- Surface acoustic wave (SAW) devices, accelerometers for mechanical properties
- Indirect optical methods using lasers, deflection techniques for displacement measurement
- Assembled micro-SEM columns to enable novel nanoscale characterization on a chip
- Assembled linear actuators to enable nanoscale testing on a chip

Advanced Electronic Interconnect

- Scanning XRD (four-circle goniometer) with area detector for simultaneous measurement and mapping of crystallographic phases, texture, and film thickness on 200 mm wafers; includes 20 μm collimation. Development is underway to make this system apply to films of thickness <10 nm, where electrical methods are invalid. Such a system can be used in-line during manufacturing.

Semiconductor Nanostructures

- Cleaving in situ within STM under ultra-high vacuum for cross-sectional imaging with atomic resolution
- Spectroscopy within STM for bandgap measurement as a function of position across a wafer
- Wafer curvature for average stress determination
- AFM-based patterned nanostructure fabrication using a nano-jet probe
- FIB nanopatterning for nanostructure position templates

Mechanical testing

- Young's modulus (E) and hardness (H) by continuous stiffness nanoindentation system and hybrid tribology/nanoindentation system; measurements still not in good agreement, with standard deviations as high as ~75 to 125 GPa for E, ~12 to 14 GPa for H for the case of thermal plasma chemical vapor-deposited (CVD) silicon carbide;
- Calibration of tip shape by use of fused quartz (not optimal)
- Nanoindentation measurements for H from sub-500 nm grains

- Modified nanoindentation measurements of sub-40 nm Si nanoparticles
- Stressed overlayer and edge lift-off tests for adhesion;
- Nanoscale scratch tests for adhesion
- Acoustic AFM for mapping of elastic properties

6. DETAILED SUMMARY OF NANOMATERIALS ISSUES

The organization of this summary follows the session titles as listed in the program. These lists were reviewed in detail at the final session of the workshop, and are sorted by topical area as presented in the workshop. There is no significance to the order in which the issues are listed.

6.1 Nanomechanical challenges and needs in manufacturing and materials processing, nanomaterial products, advanced interconnects, and active devices

6.1.1 Technological Challenges

- Each device has its own unique reliability issues
- High yield and uniformity are required
- Reliability required over 20 year lifetimes
- Acceleration factor <10 desired in order to retain relevant physical mechanisms of reliability degradation; accelerating stresses include: temperature, voltage, current, humidity, strain, vibration, laser power
- Automated assembly manufacturing unavailable for parts of size <0.5 mm; all such assembly now done by humans

6.1.2 Materials Challenges

- MEMS and NEMS:
  - Stiction (key issue)
    - Possible solution: High-stiffness designs
  - Friction and wear (sliding and impact)
    - Possible solution: Noncontacting parts
  - Hinge memory
  - Fatigue and creep
    - Possible solution: Conservative design stress
  - Stress: need to absorb, balance, control
  - Structure uniformity, defects in coatings for MEMS
  - Mechanical integrity of polysilicon vs. metals
- Carbon Nanotubes (CNTs):
  - Need dimensional uniformity, fewer defects
  - Separate by electronic structure
  - Can't make continuous fibers
  - Need much better process control and qualification
- CNTs and Nanoparticles:

  - Separation, purification methods
- Nanocomposites:
  - Need better understanding, using both simulation and experiments, especially CNT/matrix interface
- QDs:
  - Need uniform size distribution and spacing
    - Possible solution: Nanopatterning by FIB
  - Control plasticity
- General issues:
  - Make small things with more control over defects and uniformity of size
  - Structure/property relationships for monolayers (adhesion, friction, wear)
  - Effects of surface roughness
  - Develop process variables = f (residual stress, stress gradients, yield strength (YS), ultimate strength (UTS))
  - Need to know where all the atoms are located
  - Bulk property data on more materials, as foundation for nanomaterials:
    - Elastic, dielectric, density of states
  - Gradient characterization (chemical, physical), in complex matrices

6.1.3 Measurement Needs

- Tools and/or techniques to measure nanometer motion, addressing six degrees-of-freedom, faster than device resonance frequencies
  - Possible solution: optical surface profilometer, Doppler laser interferometer, strobing methods
- Tools for atomically precise engineering
  - Possible solution: Patterned ALD, using STM
- Conversion of inherently two-dimensional measurement methods to three-dimensional methods
- Metrology for micro- and nanostructure of low-level features, especially early in the process
- Tools capable of multiple, coupled measurements with a single technique
  - Thickness, crystalline phase, texture
  - These must be done quickly
  - Possible solution: scanning XRD
- Tools or systems capable of coupled measurements of properties and behaviors, using multiple techniques:
  - In- situ, coupled measurements
  - Optical coupled to mechanical methods
  - Electrical coupled to mechanical methods
- High resolution (in space and energy), fast methods for imaging, morphology, chemistry
  - Possible solution: improvements to SEM, AFM, TEM
- Methods for local measurement of:

  - Strain
  - Electrical properties
  - Structure size and/or shape
  - Temperature, including spatially resolved, non-contact
    - Possible solution: micro-Raman
- Inline-capable techniques for measurements during CMP:
  - Particle size, morphology, dispersion, aggregation, correlation to performance
- High-throughput adhesion measurement
- New tests and/or models for new material combinations
- Nondestructive measurement of defects
- Deconvolution of sample-probe interaction in nanoindentation and scanned probe microscopy (SPM)
- Standards for:
  - Reflectivity
  - film thickness and texture

6.1.4 More General Needs

- Development of relationships between in situ tests and real failures
- More modeling coupled to experimental measurements
- Large-area temperature control during fabrication to promote structure uniformity
- More collaborations to stimulate development of relevant measurement needs:
  - Manufacturing development and materials characterization/metrology development
- High-temperature interfacial reliability studies:
  - Segregation, voiding

6.2 Nanomechanical challenges and needs in characterization and testing

6.2.1 Technological Challenges

- Small length scales:
  - Difficult measurement problems even with state of the art instruments
- Need more than incremental advances in capabilities for mechanical behavior, beyond modification of existing tests, i.e., revolutionary, not just evolutionary

6.2.2 Materials Challenges

- In general (from a session announced for the Fall 2004 Materials Research Society Meeting), the dominant nanomaterial research problems at present include:
  - Size effects, temperature or viscoelasticity, multiscale modeling, adhesion/surface energy
- Need good, repeatable test samples:
  - No dislocations, surface defects

- Rapid, fully automated fabrication methods suitable for production- and research-quality specimens
- Identification of specific problems and experimental approaches applicable to nanoscale biological materials

6.2.3 Measurement Needs

- Scaling of mechanical test methods to sub micrometer samples:
  - Uniaxial, multiaxial, torsional stressing
  - Fracture toughness
  - Deformation mapping
  - Gripping, manipulation
- Localized strain measurement
- Measurement of stress gradients, especially over small dimensions
- Valid methods for separating stress and temperature effects on stress-strain behavior
- Sub micrometer temperature measurement (direct)
- Improvements in spatially resolved characterization:
  - SEM, SIMS, in-situ TEM, microdiffraction, micro-Raman, FIB (SE imaging)
  - Single-atom resolution probes such as field ion microscopes
- "Desperately needed":
  - High-resolution, *in situ* deformation studies in electron microscopes
- Nanoindentation:
  - Instrument consistency for E, H
  - Control of nanoindenter tip geometry
  - Microforce standards
  - Calibration standards for nano-test equipment
  - Direct imaging of contact areas
  - Improvements in position (x, y, z) control and measurement
- AFM:
  - Ability to do simultaneous mechanical testing and imaging
  - Ability to do simultaneous nanodeposition and imaging
    - Possible solution: dual-head AFM
  - Tip/sample interaction energies need to be understood:
    - Contact area effects
    - Direct imaging of contact areas
  - Need better characterization of:
    - AFM tips
  - Need sharp, durable, reproducible tips
  - Need AFM and model velocities to match
  - "Accurate and proven AFM technology!"
  - Better control of SPM tip motion, i.e., no unintentional lateral motion
  - Calibration standards for nano-test equipment
  - Improvements in position (x, y, z) control and measurement

- The "perfect" AFM:
  - Accurate knowledge of F and d for AFM
  - F sensor perpendicular to surface
  - Sharp, durable tip
  - F sensor with stiffness control for both mechanical properties and topography
  - Easy switch between F and d control
  - Wide range of operational frequencies, temperature
  - High-bandwidth, low-noise pre-amp
- Standards for:
  - Microforce, both axial and lateral
  - AFM spring constant
  - AFM tip-specimen contact area
  - Test/calibration specimens for nanoindentation and AFM-based mechanical testing
  - Measurement of toughness and strength of thin films
- Standard bulk property data, for which accepted measurement methods exist, should be available for all relevant compositions and processes. (Needed as a basis for understanding properties of nanomaterials):
  - Thin film and nanomaterial data should be disseminated as they become available

6.2.4 More General Needs

- MUST combine experiment and modeling for mechanical testing and adhesion/interfacial fracture.

7. BRIEF SUMMARY OF ACTION ITEMS FOR METROLOGY DEVELOPMENT

The lists of needs given above seem formidable at first glance. However, it became clear during the course of the workshop that there are several main tools commonly used by materials researchers who are concerned with understanding and improving nanomechanical reliability:

- Nanoindentation
- Micro- and nanotensile and compression testing
- Scanned probe microscopy
- Modeling

This short list of tools in no way diminishes the significance of others discussed during the workshop, e.g., electron microscopy and X-ray diffraction; it simply reflects a larger consensus about commonly used methods for nanomaterials. Attempts have been made to adapt these tools to most of the measurements listed, but considerable additional research and development are needed in order to meet the needs sufficiently. These "common-denominator" tools lead to the following action items for metrology development for nanomaterials reliability:

Near term: Improved metrological performance of scanned probe microscopy and nanoindentation, for more quantitative measurements of position, displacement, force, temperature, and other relevant quantities;

Longer term: The "atom imager," a hypothetical instrument capable of measuring nondestructively the chemical identity and precise three-dimensional position of every atom in a nanomaterial. Newer generation field ion microscopes capable of imaging with single-atom resolution are under development, but are still subject to limitations such as difficult-to-control tip (which also serves as the specimen) structure and loss of atoms upon imaging (30 to 40 % losses are not uncommon). Such instruments are also capable of sub-nanometer chemical spectroscopy.

8. GLOSSARY

AFM	Atomic force microscope, atomic force microscopy
ALD	Atomic layer deposition
CMP	Chemical mechanical polishing
CNT	Carbon nanotube
CVD	Chemical vapor deposition
d	Displacement
E	Young's modulus
F	Force
FIB	Focused ion beam
G	Strain energy release rate
H	Hardness
M	Modulus
MD	Molecular dynamics
MEMS	Micro-electromechanical system
NEMS	Nano-electromechanical system
NIST	National Institute of Standards and Technology
QD	Quantum dot
SAW	Surface acoustic wave
SE	Secondary electron
SEM	Scanning electron microscope, scanning electron microscopy
SIMS	Secondary ion mass spectroscopy
SPM	Scanned probe microscopy
STM	Scanning tunneling microscope, scanning tunneling microscopy
TEM	Transmission electron microscope, transmission electron microscopy
UTS	Ultimate tensile strength
XRD	X-ray diffraction
YS	Yield strength

ACKNOWLEDGMENTS

The purpose of this Workshop was to gather and summarize the opinions of the attendees regarding the important and timely issue of measurement needs for Reliability Issues in Nanomaterials. The organizers gratefully acknowledge the preparation; travel time; attentive, constructive, and cooperative participation; and informed opinions contributed by the attendees. Special thanks are expressed to the majority of the attendees who stayed for the summary and review of the measurement needs at the end of the workshop, as well as to those who reviewed this final report. The organizers are grateful for critical reviews of the final report, provided by the plenary speakers.

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Wendy McBride, NIST Boulder Conference Program Manager, carried out the bulk of the arrangements for this workshop. The workshop benefited substantially from the well-organized meeting room, refreshments, and al fresco lunches that Wendy arranged.

APPENDICES

The appendices to this report are intended to provide a historical record of the workshop to the extent practical. Most speakers have provided their presentations, mostly exactly as given at the workshop, some with confidential information removed. The discussions were not recorded.

Appendix A: Attendees

Arney, Susanne	Lucent Technologies Bell Labs and NJNC
Boyce, Brad	Sandia National Laboratories
Burnham, Nancy	Worcester Polytechnic Institute
Chandross, Michael	Sandia National Laboratories
Chong, Ken	National Science Foundation
Datta, Subhendu	University of Colorado
de Boer, Maarten	Sandia National Laboratories
Dunn, Marty	University of Colorado
Gerberich, William	University of Minnesota
Goldman, Rachel	University of Michigan
Greenberg, Alan	University of Colorado
Holl, Susan	Intel Corporation
Hsu, Stephen	NIST
Hurley, Donna	NIST
Johnson, Harley	University of Illinois at Urbana-Champaign
Keller, Robert	NIST
Kopycinska-Mueller, Malgorzata	NIST
Mahajan, Roop	University of Colorado
Moody, Neville	Sandia National Laboratories
Pang, Mengzhi	Cornell University
Qi, H. Jerry	University of Colorado
Randall, John	Zyvex Corporation
Read, David	NIST
Reimanis, Ivar	Colorado School of Mines
Rodbell, Ken	IBM Corporation
Roshko, Alexana	NIST
Sbaizero, Orfeo	University of Trieste (Italy)
Schwaiger, Ruth	Forschungszentrum Karlsruhe -IMF2
Sitaraman, Suresh	Georgia Institute of Technology
Smith, Douglas	NIST
Sokolov, Igor	Clarkson University
Tewary, Vinod	NIST
Uchic, Michael	Air Force Research Laboratories
Volinsky, Alex	University of South Florida
Yang, Bo	Florida Institute of Technology
Zhu, Yong	Northwestern University

Appendix B: Plenary and Breakout Presentations

Presentations from a majority of the speakers, both plenary and breakout, are included on the attached CD. For a listing of titles, please refer to appendices C and D.

Appendix C: Workshop Program

Program, Day 1

Tuesday, August 17, 2004

8:00 a.m. Registration (coffee available)

8:30 a.m. **Leslie Smith, Director, Materials Science and Engineering Laboratory, NIST** – Welcome

Topical Area: Fabrication

Fabrication I: Nanomechanical Issues in Manufacturing
Session Chair: David Read, NIST

8:45 a.m. **Susanne Arney, Lucent Technologies** – Materials Requirements and Measurements in Design and Manufacture of Micro- and Nano-Electromechanical Systems for Performance and Reliability

9:45 a.m. Break – coffee

10:00 a.m. **John Randall, Zyvex –** Materials Requirements and Measurements in Nanoscale Manufacturing and Assembly

11:00 a.m. Breakout 1:

Near-Term M/NEMS and Other Nanomaterial Products: Tools, Reliability, and Metrology
Chair: Dudley Finch, NIST/University of Colorado

12:30 p.m. Lunch (box lunch supplied)

1:30 – 2:00 p.m. Lab tours – Select ONE Tour

Tour 1. World Trade Center Investigation
Tour 2. Nanoscale Elastic-Property Imaging
Tour 3. Thin Film Mechanical Testing
Tour 4. MEMS for Testing of Biological Systems

Fabrication II: Nanomechanical Issues in Materials Processing
Session Chair: Robert Keller, NIST

2:00 p.m. **Ken Rodbell, IBM** – Materials Requirements and Measurements in Design and Manufacture of Advanced Electronic Interconnect for Performance and Reliability

3:00 p.m. Break

3:15 p.m. **Rachel Goldman, University of Michigan** – Materials Requirements and Measurements in Design and Fabrication of Semiconductor Nanostructures for Performance and Reliability

4:15 p.m. Breakout 2:

Advanced Interconnects and Active Devices: Reliability and Metrology
Chair: Alexana Roshko, NIST

5:45 p.m. Adjourn for the day

Program, Day 2

Wednesday, August 18, 2004

8:15 a.m. **Ken Chong, National Science Foundation** – NSF perspective on metrology development for nanomaterials

Topical area: Characterization and Testing

Characterization and Testing I: Mechanics of Nanomaterials
Session Chair: Roop Mahajan, University of Colorado

8:30 a.m. **William Gerberich, University of Minnesota** - Significance of Mechanical Behavior in Small Volumes to Design of Reliable Nanomaterials and Nanoscale Devices

9:30 a.m. Break

9.45 a.m. **Martin Dunn, University of Colorado** - Necessary Reinterpretations of Continuum Mechanics Concepts for Applicability to Nanomaterials

10:45 a.m. Breakout 3:

Unusual Mechanical Properties of Nanomaterials
Chair: Martin Dunn, University of Colorado

12:15 p.m. Lunch (box lunch supplied)

1:15 – 1:45 p.m. Lab tours – Select ONE Tour

Tour 1. World Trade Center Investigation
Tour 2. Nanoscale Elastic-Property Imaging
Tour 3. Thin Film Mechanical Testing
Tour 4. MEMS for Testing of Biological Systems

Characterization and Testing II: Mechanical Probing Methods for Characterizing Nanomaterials
Session Chair: Donna Hurley, NIST

1:45 p.m. **Nancy Burnham, Worcester Polytechnic Institute** – Role of Atomic Force Microscopy in Conception, Design, and Characterization of High-Performance and Reliable Nanomaterials

2:45 p.m. Break

3:00 p.m. **Neville Moody, Sandia National Laboratories** – Significance of Adhesion and Interfacial Fracture in Design of Reliable Nanomaterials and Nanoscale Devices

4:00 p.m. Breakout 4:

Atomic Force Microscopy and Nanoindentation
Chair: Ken Gall, University of Colorado

5:30 p.m. Adjourn technical sessions for day 2

5:45 p.m. Board bus for travel to Flagstaff Summit

6:00 p.m. Picnic, Flagstaff Stone Shelter (casual clothing suggested)

Program, Day 3

Thursday, August 19, 2004

Topical area: Nanomaterials metrology roadmap

Session Chair: Stephen Hsu, NIST

8:30 a.m.	**Stephen Hsu, National Institute of Standards and Technology** – The Nanomechanics Measurement Facility in the NIST Advanced Measurement Laboratory (AML)
8:45 a.m.	**Douglas Smith, National Institute of Standards and Technology** – Nanomechanics at NIST
9:45 a.m.	Break
10:00 a.m.	**Robert Keller, National Institute of Standards and Technology** – Summary of nanomaterials reliability issues and measurement needs
11:00 a.m.	**David Read, National Institute of Standards and Technology** – Challenges, barriers, and priorities for nanomaterials metrology
12:00 noon	Adjournment of the technical sessions of the workshop
1:00 p.m.	Lab tours at University of Colorado, by request
1:00-5:00 p.m.	Report writing by volunteers

Appendix D: Breakout Presentation Titles

Breakout 1: Near-Term M/NEMS and Other Nanomaterial Products: Tools, Reliability, and Metrology

Chair: Dudley Finch

DeBoer, Maarten	*In situ* Property Measurement in MEMS
Greenberg, Alan	Mechanical and Gas Transport Behavior of Dense Polymer Films
Sbaizero, Orfeo	The Center for Excellence in Nanotechnology at the University of Trieste
Volinsky, Alex	Thin Film Mechanical Reliability: Environmental Effects

Breakout 2: Advanced Interconnects and Active Devices: Reliability and Metrology

Chair: Alexana Roshko

Sitaraman, Suresh	Off-Chip Interconnects: Design, Fabrication, and Reliability
Johnson, Harley	Mechanical and Electronic Coupling in Nanotubes and Quantum Dots
Yang, Bo	Energy Release Rate of Quantum Islands in Stranski-Krastanow Growth
Holl, Susan	CMOS to beyond CMOS, Novel Material Characterization Challenges
Pang, Mengzhi	Adhesion Measurements of Thin Film Structures

Breakout 3: Unusual Mechanical Properties of Nanomaterials

Chair: Martin Dunn

Uchic, Michael	Mechanical Testing at the Micron-Size Scale
Schwaiger, Ruth	Investigations of Nanostructured Materials – from Nanocrystalline to Nanoporous
Datta, Subhendu	Measurement and Modeling of Thermomechanical Properties of Thin Layers
Qi, H. Jerry	Biomolecule Unfolding and Mechanics of Nacre
Reimanis, Ivar	Toughness Measurement of Thin Films: What Does it Tell Us and How Reliable is That?
Boyce, Brad	Measurement Challenges in Microtensile and Microfatigue Reliability Studies

Breakout 4: Atomic Force Microscopy and Nanoindentation

Chair: Ken Gall

Sokolov, Igor	The Need in a Standard for Small Force to Probe Interactions in Nanoscale with the AFM
Zhu, Yong	Novel Experiments to Assess the Mechanical Properties of Thin Films and Nano-Scale Structures
Chandross, Michael	Simulations of Nanotribology

Appendix E: Questions Posed to Plenary Session Speakers

Broad questions for all Plenary speakers; treat in terms of your area of expertise:

- What is considered state-of-the-art, in terms of experiment/processing/characterization **and/or** theory/modeling? (This may include some of your own work.)
- What are the problems now under investigation?
- What barriers (scientific and/or technological) do you foresee to advances in the coming 5 to 10 years? Please include barriers that may be addressed by improvements in materials measurements.
- What are possible approaches to solution? Please include how you might envision "division of labor," *i.e.*, roles of industry, DOE labs, NIST, universities.

Questions for Fabrication speakers (Day 1, Arney, Randall, Rodbell, Goldman):

- How is mechanics/mechanical behavior important in terms of performance and reliability required at product level?
- How do you design-in performance and reliability, using principles of micro- and nanostructure control? Please comment on adequacy of existing lifetime or performance prediction methods.
- How does industry measure performance of both the end product and the constituent materials?
- What are the limitations/barriers of the present measures/metrology?

Questions for Characterization and Testing speakers (Day 2, Gerberich, Dunn, Burnham, Moody):

- Measurements of which properties do you foresee to be critical for advancing implementation of nanomaterials in the coming decade?
- How sufficient are the existing test methods for nanoscale property measurement? Can advances be made incrementally, or are revolutionary changes needed?
- Please comment on the role of modeling in your work. What answers do you seek? What type of modeling do you perform or refer to?
- What kind of information about materials structure is needed to guide the development of property characterization and testing methods?

Appendix F: Questions Posed to Breakout Session Speakers

1. How does metrology affect your present work in nanomaterials?

2. What questions relating to reliability of nanomaterials would you like to see addressed in this workshop and report/roadmap?

3. What metrology issues are of the most significance to you for your work in the coming 5 to 10 years?

Appendix G: NIST Lab Tours

Tour 1. World Trade Center investigation – David McColskey

In August of 2002, NIST took responsibility for the investigation of the World Trade Center disaster. The investigation objectives include determining why the buildings collapsed; the procedures and practices used in the design, construction, operation, and maintenance of the buildings; and areas in codes and practices that warrant revision. The eight interdependent projects include Project 3, "Analysis of Structural Steel," led by the Materials Science and Engineering Laboratory. The objective of the project is to "determine and analyze the mechanical and metallurgical properties and quality of steel, weldments, and connections from steel recovered from WTC 1, 2, and 7."The lab tour/presentation will take place in the Mechanical Properties Lab, where many specimens of steels from structural components of the WTC towers are stored and evaluated.

Tour 2. Nanoscale Elastic-Property Imaging – Donna Hurley

We are developing AFM-based metrology for rapid, nondestructive measurement of mechanical properties with true nanoscale spatial resolution. Our hybrid acoustic/AFM approach involves the vibrational resonance of an AFM cantilever when its tip is in contact with a sample. We have demonstrated how this approach can be used to determine the local indentation modulus of an unknown material or thin film. The AFM's scanning capabilities mean that we can also obtain in-situ images of relative elastic stiffness with nanoscale spatial resolution. Current research is aimed at combining the quantitative, single-point methods with qualitative imaging in order to obtain quantitative maps of nanomechanical properties. Complementary information obtained with scanning electron microscopy provides insight into structure-property relations and helps to interpret the nanoscale contact-mechanics behavior.

Tour 3. Thin Film Mechanical Testing – David Read

We perform microtensile testing on thin films of metals, polymers, and ceramics. A typical gage section is 1 μm thick by 10 μm wide by 200 μm long. The specimen is patterned by

photolithography on bare silicon. The silicon is etched from beneath the specimen, to a depth of at least 60 μm, using xenon difluoride. This etch frees the gage section and a grip section with a hole for the loading pin. Tests are done under an optical microscope or inside the SEM. Using a three-axis micromanipulator, the loading pin is placed in the 50 μm diameter hole in the grip section of the specimen. As the load is applied, the displacement of a calibrated spring is recorded. A calibration factor, obtained using a force pendulum, is used to convert the measured displacements to force values. Our noise level is tens of micronewtons; failure loads for our specimens range upward from around 1 mN. Digital images are recorded every few seconds during the test; digital image correlation is used to obtain the engineering strain in the gage section. The gage section of the specimen is imaged over about 800 pixels of the digital images, and the displacements are obtained with a precision of 0.01 to 0.05 pixels, for a strain noise level of about 60 microstrain. Data are presented in the form of engineering stress-strain curves. Visitors to the lab will be able to see the optical testing station, the micromanipulator, the xenon difluoride etching system, and posters showing typical specimens and results.

Tour 4. MEMS for Testing of Biological Systems – Andrew Slifka

We are developing MEMS-based mechanical test platforms and tools that can be integrated with currently used biological techniques for the evaluation and measurement of cellular response (*e.g.*, gene expression, cell morphology, area of adhesion, etc.). These types of studies are needed because the development of vascular smooth muscle cells in cardiovascular tissue, for example, is dependent on variations in the stress-strain environment that result from the expansion and contraction of the vessel wall. The importance of the environment becomes apparent when one considers that engineered tissues have mechanical properties inferior to those of naturally grown tissues. This is possibly a bulk effect, but is clearly related to processes at the cellular level. Without a quantitative understanding of the mechanics and functionality of the building blocks (cells and fibers), the bulk properties of the tissues cannot be fully understood and modeled.

NIST Technical Publications

Periodical

Journal of Research of the National Institute of Standards and Technology—Reports NIST research and development in metrology and related fields of physical science, engineering, applied mathematics, statistics, biotechnology, and information technology. Papers cover a broad range of subjects, with major emphasis on measurement methodology and the basic technology underlying standardization. Also included from time to time are survey articles on topics closely related to the Institute's technical and scientific programs. Issued six times a year.

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